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IS 7299 (1974): Mineral Oil for Cosmetic Industry [PCD 19: Cosmetics]



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IS : 7299 - 1974
(Reaffirmed 2006)

Indian Standard
SPECIFICATION FOR
MINERAL OIL FOR COSMETIC INDUSTRY

Second Reprint JULY 2007
(Including Amendment Nos. 1, 2 & 3)

UDC 668.58 : 665.6.7

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

AMENDMENT NO. 3 FEBRUARY 1983
TO
IS:7299-1974 SPECIFICATION FOR MINERAL OIL FOR
COSMETIC INDUSTRY

Alteration

(Page 10, clause A-5.2.2, line 3) - Substitute
'0.004 mg' for '0.004 g'.

(PCDC 19)

AMENDMENT NO. 2 MARCH 1982

TO

IS:7299-1974 SPECIFICATION FOR MINERAL
OIL FOR COSMETIC INDUSTRY

Alteration

(Page 9, clause A-4.2.3, line 3) - Substitute
'1 000 ml of solution' for '100 ml of solution'.

(PCDC 19)

AMENDMENT NO. 1 DECEMBER 1977
TO
IS : 7299-1974 SPECIFICATION FOR MINERAL OIL
FOR COSMETIC INDUSTRY

Alterations

(*Page 5, Table 1, col 3*)

a) *Against Sl No. (iii)* — Substitute the following for the existing matter:

8	to	30	}	†
31	to	63		
64	and above			

b) *Against Sl No. (iv)* -- Substitute the following for the existing matter:

0.815 to 0.910†

c) *Against Sl No. (xii)* — Substitute '0.1' for '0.80'.

†For any specific value within the range, it shall be as agreed to between the purchaser and the supplier.

(CDC 39)

Indian Standard

SPECIFICATION FOR MINERAL OIL FOR COSMETIC INDUSTRY

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(Continued on page 2)

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IS : 7299 - 1974

(Continued from page 1)

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Indian Standard

SPECIFICATION FOR MINERAL OIL FOR COSMETIC INDUSTRY

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 13 March 1974, after the draft finalized by the Cosmetics and Toilet Goods Sectional Committee had been approved by the Chemical Division Council.

0.2 Mineral oil finds use in the formulation of many cosmetics and toilet goods. It possesses excellent stability to heat, light, acids and alkalis, has no colour or odour and is harmless to skin.

0.3 In the preparation of this standard assistance has been derived from the following publications:

TPF Specification No. 1A Mineral oil. Toilet Preparations Federation Ltd, UK.

TGA Specification No. 1 Mineral oil. Toilet Goods Association Inc, USA.

British Pharmacopoeia. 1968. General Medical Council, London.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes requirements and methods of sampling and test for mineral oil for cosmetic industry.

2. TYPES

2.1 There shall be three types of mineral oil, namely, light, medium and heavy.

*Rules for rounding off numerical values (*revised*).

IS : 7299 - 1974

3. REQUIREMENTS

3.1 Description — The material shall consist wholly of highly refined mineral oil, free from turbidity, sediment, water, foreign matter and visible impurities. It shall be colourless, tasteless, odourless and free from fluorescence. The material shall be insoluble in water and alcohol and soluble in ether and chloroform.

3.2 Colour — The colour of the oil shall be not inferior than $1.2Y + 0.3R$ Lovibond units when measured in 18 inch cell according to the method prescribed in IS : 1448 [P:13] - 1960* or +25 Saybolt units according to the method prescribed in IS : 1448 [P:14]-1967†.

Alternatively, the colour of the oil shall be as agreed to between the purchaser and the supplier.

3.3 The material shall also comply with the requirements given in Table 1 when tested according to the method referred to in col 4 and 5 of the table.

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in suitable containers as agreed to between the purchaser and the supplier.

4.2 Marking — Each container shall be suitably marked with the following information:

- a) Name and type of the material,
- b) Colour of the oil in Lovibond or Saybolt units,
- c) Certificate that the oil passes the UV absorption test,
- d) Name of the manufacturer,
- e) Batch or code number, and
- f) Volume of contents.

4.2.1 The product may also be marked with Standard Mark.

4.2.2 The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufactures or producers may be obtained from the Bureau of Indian Standards.

*Methods of test for petroleum and its products, P: 13 Colour by Lovibond tintometer.

†Methods of test for petroleum and its products, P: 14 Colour by Saybolt chromometer (first revision).

TABLE 1 REQUIREMENTS FOR MINERAL OIL FOR COSMETIC INDUSTRY

(Clause 3.3)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO	
			Cl No. of Appendix A	' P ' of IS : 1448*
(1)	(2)	(3)	(4)	(5)
i)	Cloud point, °C, <i>Max</i>	5·0	—	P : 10
ii)	Odour:			
	a) at room temperature	None	—	—
	b) when heated at 95 to 98°C for $\frac{1}{2}$ h in a water-bath	Not objectionable	—	—
iii)	Viscosity at 37·8°C, centistokes:		—	P : 25
	Light	8 to 30		
	Medium	31 to 63		
	Heavy	64 and above		
iv)	Relative density at 25°C/25°C	0·815 to 0·910	—	P : 32
v)	Free acid and alkali	To pass the test	A-2	—
vi)	Saponification value	Nil	—	P : 55
vii)	Sulphur and sulphides	To pass the test	A-3	—
viii)	Carbonizable substances	To pass the test	A-4	—
ix)	Ash, percent by mass, <i>Max</i>	0·01	—	P : 4
x)	Arsenic (as As_2O_3), parts per million, <i>Max</i>	2·0	A-5	—
xi)	Lead, parts per million, <i>Max</i>	20	A-6	—
xii)	Ultra violet absorption test, extinction, <i>Max</i>	0·80	A-7	—
xiii)	Stability test	do	A-8	—

*Methods of test for petroleum and its products:

IS : 1448 (P : 4)-1968 Ash, sulphated ash and water soluble ash (*first revision*).IS : 1448 (P : 10)-1970 Cloud point and pour point (*first revision*).

IS : 1448 (P : 25)-1960 Kinematic viscosity.

IS : 1448 (P : 32)-1972 Density and relative density (*first revision*).

IS : 1448 (P : 55)-1963 Saponification value, saponifiable and unsaponifiable matter.

5. SAMPLING

5.1 Representative test samples of the material shall be drawn as prescribed in IS : 1447-1966*.

5.2 Number of Tests — Test for all the characteristics given in 3 shall be carried out on the composite sample.

5.3 Criteria for Conformity — The material shall be taken to have conformed to this specification if the composite sample satisfies all the requirements.

A P P E N D I X A

(Clause 3.3, and Table 1)

METHODS OF TEST FOR MINERAL OIL FOR COSMETIC INDUSTRY

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960†) shall be used in tests.

NOTE — ‘ Pure chemicals ’ shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. TEST FOR FREE ACID AND ALKALI

A-2.1 Procedure — Shake 20 g of the material with an equal amount of hot distilled water. Test the aqueous portion with blue litmus and red litmus.

A-2.1.1 The material shall be taken to have passed the test if neither blue litmus nor red litmus changes colour.

A-3. TEST FOR SULPHUR AND SULPHIDES

A-3.1 Reagent

A-3.1.1 Copper Strips — 1 cm in width and freshly polished.

A-3.2 Procedure — Place in a beaker about 100 g of the sample and keep on a water-bath at a temperature of 95°C. Then place a strip of copper in

*Method of sampling of petroleum and its products.

†Specification for water, distilled quality (*revised*).

the sample so that it is partially immersed in it and allow to remain for 10 minutes.

A-3.2.1 The material shall be taken to have passed the test if the copper strip used in the test shows no tarnishing when compared with another freshly polished copper strip.

A-4. TEST FOR CARBONIZABLE SUBSTANCES

A-4.1 Apparatus

A-4.1.1 Test Tubes — As shown in Fig. 1, of heat-resistant glass fitted with a well-ground glass stopper, the stopper and the tube bearing identical and indestructible numbers. The tube shall be 140 ± 3 mm in length and 14 ± 1 mm in outside diameter, and shall be calibrated at the 5 ± 0.2 ml and 10 ± 0.2 ml liquid levels. The capacity of the tube with stopper inserted shall be 16 ± 1.0 ml. A rolled edge may be provided for suspending the tube on the cover of the water bath.

A-4.1.2 Water-Bath — A water-bath suitable for immersing the test tube above the 10 ml line and equipped to maintain a temperature of $100 \pm 0.5^\circ\text{C}$. The bath shall be provided with a cover of any suitable material with holes approximately 16 mm in diameter through which the test tubes may be suspended.

A-4.1.3 Colour Comparator — A colour comparator, of suitable type for observing the colour of the acid layer in comparison with the reference standard colour solution. The size and shape of the comparator are optional but the size and shape of the apertures shall conform to the dimensions prescribed in Fig. 1.

A-4.2 Reagents

A-4.2.1 Concentrated Sulphuric Acid — nitrogen-free, which is tested as follows:

Dilute a small amount of the acid with an equal volume of water and superimpose 10 ml of the cooled liquid upon diphenylamine solution (1 g of diphenylamine in 100 ml of concentrated sulphuric acid). Blue colour should not appear at the zone of contact within 1 hour.

A-4.2.2 Cobaltous Chloride Solution — 0.5 N. Dissolve about 65 g of cobaltous chloride ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$) in enough dilute hydrochloric acid (1 : 39) to make 1 000 ml of solution. Transfer exactly 5 ml of this solution to a flask; add 16 ml of sodium hydroxide solution (1 : 5) and 5 ml of hydrogen peroxide. Boil for 10 minutes, cool and add 2 g of potassium iodide and 20 ml of sulphuric acid (1 : 4). When the precipitate has dissolved, titrate the liberated iodine with 0.1 N sodium thiosulphate solution using starch as indicator. Each millilitre of sodium thiosulphate solution consumed is

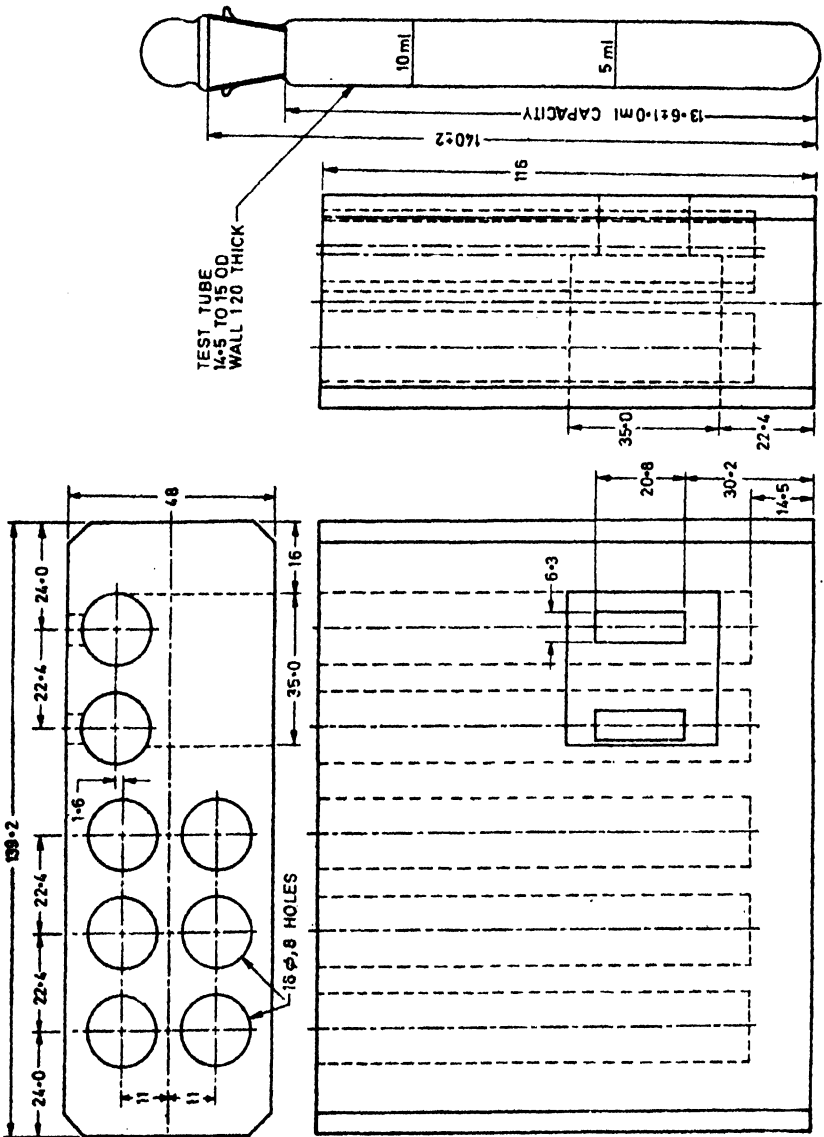


FIG. 1 COLOUR COMPARATOR FOR TEST FOR CARBONIZABLE SUBSTANCES

All dimensions in millimetres.

equivalent to 0.023 799 g of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$. Adjust the final volume of cobalt chloride solution by the addition of dilute hydrochloric acid (1 : 39) so that 1 ml contains 59.5 mg of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$.

A-4.2.3 Ferric Chloride Solution — 0.5 N. Dissolve about 55 g of ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) in enough diluted hydrochloric acid (1 : 39) to make 100 ml of solution. Transfer exactly 10 ml of the solution to a flask, add 5 ml of concentrated hydrochloric acid, 25 ml of water and about 3 g of potassium iodide. Stopper and allow the mixture to stand for 5 minutes. Dilute the mixture with 50 ml of water and titrate the liberated iodine with 0.1 sodium thiosulphate solution using starch solution as indicator. Each millilitre of 0.1 N thiosulphate solution is equivalent to 0.027 03 g of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$. Adjust the final volume of ferric chloride solution by addition of dilute hydrochloric acid (1 : 39) so that 1 ml contains 45.0 mg of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$.

A-4.2.4 Cupric Sulphate Solution — 0.5 N. Dissolve about 65 g of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in enough dilute hydrochloric acid (1 : 39) to make 1 000 ml of solution. Transfer exactly 10 ml of this solution to a flask, add 50 ml of water, 4 ml of acetic acid, and 3 g of potassium iodide. Allow the mixture to stand for 5 minutes, then titrate the liberated iodine with 0.1 N thiosulphate solution using starch solution as indicator. Each millilitre of 0.1 N thiosulphate solution is equivalent to 0.024 97 g of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$. Adjust the final volume of copper sulphate solution by the addition of diluted hydrochloric acid (1 : 39) so that 1 ml contain 62.4 mg $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$.

A-4.2.5 Reference Colorimetric Solution — Prepare a reference standard pale amber solution for colour comparison by mixing together 1.5 parts of cobalt chloride solution, 3.0 parts of the ferric chloride solution and 0.5 parts of the copper sulphate solution. Measure 5 ml of this mixture into a test tube as specified in A-4.1.1. This pale amber reference standard shall then be overlaid with 5 ml of mineral oil.

A-4.3 Procedure

A-4.3.1 Clean a test tube with a chromic acid cleaning solution, rinse with tap water followed by distilled water, and dry in an oven at 105°C for 1 hour.

A-4.3.2 Fill the test tube to the 5 ml mark with sulphuric acid (94.7 ± 0.2 percent). Then add the oil to be tested to the 10 ml mark, insert the stopper loosely, and place the test tube in position in the water-bath at $100 \pm 0.5^\circ\text{C}$.

A-4.3.3 After the test tube has been in the water-bath for 30 seconds, loosen the stopper sufficiently to release any pressure and reinsert, remove the test tube from the bath quickly, hold with a finger over the stopper, and give three vigorous, vertical shakes over an amplitude of about 12 cm, shaking the test tube quickly and at a rate corresponding to 5 shakes per second. Repeat every 30 seconds. Do not keep the test tube out of the bath longer than 3 seconds for each shaking period.

A-4.3.4 At the end of 10 minutes from the time the test tube was first placed in the bath, remove the test tube and allow to stand in the room for not less than 10 minutes nor more than 30 minutes. Note any discolouration of the oil layer. Place the test tube in the colour comparator and compare the acid layer with 5 ml of the standard colorimetric solution and 5 ml of the sample mineral oil in the test tube that has been shaken vigorously for 10 seconds and allowed to stand just long enough for the contents to separate into two layers.

A-4.3.5 The mineral oil shall be reported as passing the test only when the oil layer shows no change in colour and when the acid layer is not darker than the reference standard colorimetric solution.

NOTE — A bluish haze in the oil layer should not be interpreted as a change in colour.

A-4.3.5.1 If the oil layer is discoloured or if the acid layer is darker than the reference standard colorimetric solution, the mineral oil shall be reported as not passing the test.

A-5. TEST FOR ARSENIC

A-5.1 Reagents

A-5.1.1 *Concentrated Sulphuric Acid*

A-5.1.2 *Concentrated Nitric Acid*

A-5.2 Procedure

A-5.2.1 *Preparation of Sample* — Weigh 2.000 g of the sample in a Kjeldahl flask of 500 ml capacity. Add 15 ml of concentrated sulphuric acid followed by 4 ml of concentrated nitric acid. Heat cautiously. Add drop by drop more nitric acid, if required, from a pipette to speed up the oxidation of the sample. The total amount of nitric acid shall be noted for use in the control test. When oxidation is complete, the solution is clear and faint yellow; at that stage, add 20 ml of water and again boil to fuming. Ensure removal of all nitric acid.

A-5.2.2 Carry out the test for arsenic with the solution prepared in **A-5.2.1** as described in IS : 2088-1962*. Compare the stain obtained with that produced with 0.004 g of arsenic trioxide.

A-6. TEST FOR LEAD

A-6.1 Apparatus

A-6.1.1 *Nessler Cylinders* — 50 ml capacity, matched.

A-6.2 Reagent

A-6.2.1 *Ammonium Acetate Solution* — 10 percent

*Modified Gutzeit method of test for arsenic.

A-6.2.2 Ammonium Citrate Solution — Dissolve 8.75 g of citric acid in water, neutralize with ammonia and dilute with water to 100 ml.

A-6.2.3 Ammonium Hydroxide — 10 percent (m/m).

A-6.2.4 Potassium Cyanide Solution — 10 percent.

A-6.2.5 Sodium Sulphide Solution — 10 percent.

A-6.2.6 Standard Lead Solution — Dissolve 1.600 g of lead nitrate in water, and 10 ml of concentrated nitric acid and dilute to 1 000 ml. Pipette out 10 ml of the solution and dilute it again to 1 000 ml with water. One millilitre of the final solution contains 0.01 mg of lead (as Pb). The solution should be freshly prepared.

A-6.3 Procedure

A-6.3.1 Preparation of Sample — Treat 2.000 g of the sample as prescribed in A-5.2.1.

A-6.3.2 Take the solution prepared in A-6.3.1 in a Nessler cylinder, add 10 ml of ammonium acetate solution, 5 ml of ammonium citrate solution, 5 ml of ammonium hydroxide and 1 ml of potassium cyanide solution and dilute to 50 ml with water; then add two drops of sodium sulphide solution and mix well. In another Nessler cylinder, carry out a control test using 1 ml of standard lead solution and the same quantities of other reagents as used in the test with the material.

A-6.3.3 The material shall be taken as not having exceeded the limit prescribed in Table 1 if the intensity of colour produced with the material is not greater than that produced in the control test.

A-7. DETERMINATION OF ULTRA VIOLET ABSORPTION

A-7.0 General — The extent to which radiation is absorbed in passing through a layer of an absorbing substance is expressed in terms of the extinction, E . This purely optical quantity is defined by the expression:

$$E = \log_{10} (I_0/I)$$

where I_0 is the intensity of the radiation passing into the absorbing layer and I the intensity of the radiation passing out of it. For a solution of an absorbing solute contained in an absorption cell having flat parallel optical faces, results are evaluated by the expression:

$$E_{1 \text{ percent, } 1 \text{ cm}} = E/c \cdot l$$

where c is the concentration of absorbing solute expressed as a percentage (m/v) and l the thickness of the absorbing layer in centimetre. Therefore, $E_{1 \text{ percent, } 1 \text{ cm}}$ is the extinction of a 1-cm layer of 1 percent (m/v) solution of the absorbing solute, its value at a particular wavelength in a given solvent being a property of the solute.

A-7.1 Procedure

A-7.1.1 In measuring the extinction of a solution at a given wavelength, the extinction of the solvent cell and its contents shall not exceed 0.4 per cm of path length, and shall in general be less than 0.2 per cm of path length, when measured with reference to air at the same wavelength. The solvent in the solvent cell shall be of the same batch as that used to prepare the solution and shall be free from fluorescence at the wavelength of measurement. Statements of concentration and thickness of the solution to be used in the determination of the light absorption apply to measurements made with photoelectric instruments. When such details are not given or when measurements are made by a photographic or visual instruments, the concentration and thickness of solution shall be adjusted so that the measured extinction lies within the optimum working range of the apparatus.

A-7.1.2 For this test it is more convenient to use a recording instrument, a 1-cm layer of 2.0 percent *m/v* solution in trimethyl pentane in the wavelength range of 240 to 280 μm . If these conditions are not appropriate for a particular instrument, the thickness should be varied and not the concentration.

A-7.1.3 When measuring the extinction of an absorption medium the spectral slit width shall be small compared with the half width of the absorption band, otherwise erroneously low extinctions will be measured. Particular care is needed in this respect and the instrumental slit width used should always be such that a further reduction does not result in an increased extinction reading.

A-8. STABILITY TEST

A-8.1 Apparatus

A-8.1.1 *Ultra Violet Lamp*

A-8.2 Procedure — Take 50 ml of the material in a clean beaker, place under the ultra violet lamp and expose it thus for a total period of 6 hours. Measure the colour of the oil, either in Lovibond units using 18 inch cell according to method prescribed in IS : 1448 [P:13] - 1960* or in Saybolt units according to method prescribed in IS : 1448 [P:14] - 1967†.

A-8.2.1 The material shall be taken to have passed the test if the colour of the oil is not darker than:

- a) 1.2Y + 0.3R Lovibond units,
- or
- b) +25 Saybolt units.

*Methods of test for petroleum and its products, P: 13 Colour by Lovibond tintometer.

†Methods of test for petroleum and its products, P: 14 Colour by Saybolt chromometer (first revision).

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GUWAHATI 781003

245 6508

5-8-56C, L.N. Gupta Marg, Nampally Station Road, HYDERABAD 500001

2320 1084

Prithvi Raj Road, Opposite Bharat Overseas Bank, C-Scheme, JAIPUR 302001

222 3282

11/418 B, Sarvodaya Nagar, KANPUR 208005

223 3012

Sethi Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road,
LUCKNOW 226001

261 8923

H. No. 15, Sector-3, PARWANOO, Distt. Solan (H.P.) 173220

235 436

Plot No A-20-21, Institutional Area, Sector 62, Goutam Budh Nagar, NOIDA 201307

240 2206

Patliputra Industrial Estate, PATNA 800013

226 2808

Plot Nos. 657-660, Market Yard, Gultkdi, PUNE 411037

2427 4804

Sahajanand House 3rd Floor, Bhaktinagar Circle, 80 Feet Road,
RAJKOT 360002

237 8251

T.C. No. 2/275 (1 & 2), Near Food Corporation of India, Kesavadasapuram-Ulloor Road,
Kesavadasapuram, THIRUVANANTHAPURAM 695004

255 7914

1st Floor, Udyog Bhavan, VUDA, Siripuram Junction, VISHAKHAPATNAM-03

271 2833

*Sales Office is at 5 Chowringhee Approach, P.O. Princep Street, KOLKATA 700072

2355 3243

†Sales Office (WRO) Plot No. E-9, MIDC, Rd No. 8, Behind Telephone Exchange,
Andheri (East), Mumbai-400 0093

2832 9295